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1,1′-(Ethane-1,2-diyl)dipyridinium dibromidodichloridomercurate(II)

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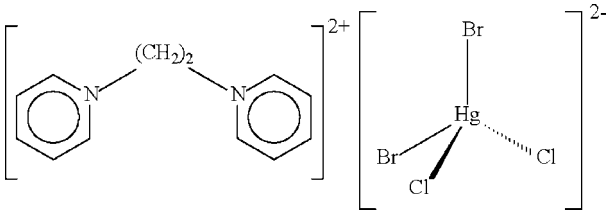
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.009 Å; disorder in main residue; R factor = 0.034; wR factor = 0.102; data-to-parameter ratio = 17.1.

The Hg atom in the title compound, $(C_{12}H_{14}N_2)[HgBr_2Cl_2]$, is coordinated by four halogen atoms in a tetrahedral geometry. The cation lies on a centre of inversion and the anion about a mirror plane. The two halogen atoms on the mirror plane are disordered between Br and Cl in ratios of 0.921 (4):0.079 (4) and 0.795 (4):0.205 (4). The halogen atom in the general position is disordered between Br and Cl in a ratio of 0.142 (1):0.858 (1).

Related literature

For ammonium salts of tetrahalidomercurates(II), see: Amami *et al.* (2002); Kamenar & Nagl (1976); Pakhomov *et al.* (1991); Pickardt *et al.* (2006). For the synthesis of the organic component, see: Xiao *et al.* (2006).



Experimental

Crystal data

$(C_{12}H_{14}N_2)[HgBr_2Cl_2]$
M_r = 617.56
Orthorhombic, *Pnma*
a = 17.952 (1) Å
b = 14.381 (1) Å
c = 6.6707 (5) Å
V = 1722.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 13.88 mm^{-1}
T = 298 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.168, T_{max} = 0.338
(expected range = 0.124–0.250)
9021 measured reflections
1572 independent reflections
1395 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.102
S = 1.05
1572 reflections
92 parameters
57 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.40 e Å^{-3}
$\Delta\rho_{min}$ = −1.17 e Å^{-3}

Table 1
Selected bond lengths (Å).

Hg1—Cl2	2.37 (11)	Hg1—Br2	2.548 (3)
Hg1—Br1	2.41 (5)	Hg1—Cl1	2.571 (19)
Hg1—Cl3	2.50 (3)	Hg1—Br3	2.584 (3)

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2386).

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supplementary materials

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1,1'-(Ethane-1,2-diyl)dipyridinium dibromidodichloridomercurate(II)

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Comment

The discrete tetrahedral tetrahalogenomercurate(II) dianion is characterized in a number of salts (Allen, 2002; CSD Version 5.28); examples of the ammonium salts include, for example, bis(tetramethylammonium) tetrachloromercurate (Amami *et al.*, 2002), bis(tetramethylammonium) tetrabromomercurate (Kamenar & Nagl, 1976) and bis(tetramethylammonium) tetraiodomercurate (Pakhomov *et al.*, 1991; Pickardt *et al.*, 2006). The mercury atom in $[C_{12}H_{14}N_2][HgBr_2Cl_2]$ is coordinated by four halogen atoms in a tetrahedral geometry; the halogen atoms are disordered (Fig. 1). Selected bond distances are given in Table 1.

Experimental

The salt was synthesized from the reaction of ethane-1,2-dipyridinium dibromide (0.035 g, 0.1 mmol) in methanol (5 ml) and mercuric chloride (0.054 g, 0.2 mmol) in DMF (10 ml). The mixture was set aside for the formation of colorless needle-shaped crystals in 40% yield after several days. A needle was cut to give a columnar specimen. The organic reactant was synthesized by using a literature method (Xiao *et al.*, 2006).

Refinement

Of the three halogens in the asymmetric unit, one lies in a general position and the other two on a mirror plane. Initial attempts to refine the structure with either three chlorines or three bromines gave unacceptably high *R*-indices and large peaks/deep holes. The three halogen atoms were then refined as three (Br+Cl) mixtures; one attempt allowed the mixtures to have the same displacement parameters as well as sharing the same site. Another attempt had the components having the same displacement parameters only. The second led to a formulation consisting of approximately two Br and two Cl atoms. The use of a restraint that fixed the number of Br and Cl atoms as exactly two each led to occupancies of 0.142 (1), 0.921 (4) and 0.795 (4), respectively, for Br1, Br2 and Br3, and 0.858 (1), 0.079 (4) and 0.205 (4), respectively, for Cl1, Cl2 and Cl3. The formulation is in fair agreement with CH&N elemental analysis.

Disorder also affected the cation; the pyridyl ring was refined as a rigid hexagon (C—C = C—N = 1.39 Å). The C(sp^3)—C(sp^3) distance was restrained to 1.50 (1) Å, and the N···C(sp^3) distance to 2.45 (1) Å. The displacement parameters of atoms of the cation were restrained to be nearly isotropic. C-bound H atoms were positioned geometrically (C–H = 0.93 and 0.97 Å), and were included in the refinement in the riding model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$.

The final difference Fourier map had a large peak at 0.89 Å from N1, indicating a possible disorder in the cation. But no suitable disorder model was found for the cation.

Figures

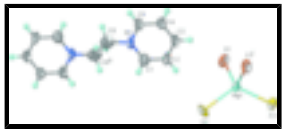

Fig. 1. The molecular structure of $[C_{12}H_{14}N_2][HgBr_2Cl_2]$, with displacement ellipsoids drawn at the 50% probability level. The bromine and chlorine atoms are disordered; the figures depicts the anion as an $[HgX_4]^{2-}$ species. The halogen atom in the general position is labelled X1 (and the symmetry-related $X1^{i}$); those in the special position are labelled X2 and X3. Hydrogen atoms are drawn as spheres of arbitrary radius. [Symmetry code (i): x, 3/2 – y, z.]. Unlabelled atoms in the cation are related to the labelled ones by the symmetry code (1 – x, 1 – y, 1 – z).

1,1'-(Ethane-1,2-diyl)dipyridinium dibromidodichloridomercurate(II)

Crystal data

$(C_{12}H_{14}N_2)[HgBr_2Cl_2]$	F_{000} = 1136
M_r = 617.56	D_x = 2.382 Mg m^{-3}
Orthorhombic, *Pnma*	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2n	Cell parameters from 2764 reflections
a = 17.952 (1) Å	θ = 2.3–22.7º
b = 14.381 (1) Å	μ = 13.88 mm^{-1}
c = 6.6707 (5) Å	T = 298 (2) K
V = 1722.1 (2) Å^3	Column, colourless
Z = 4	0.20 × 0.10 × 0.10 mm

Data collection

Bruker APEX area-detector diffractometer	1573 independent reflections
Radiation source: fine-focus sealed tube	1395 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.038
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.3º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −21→20
T_{min} = 0.168, T_{max} = 0.338	k = −16→17
9021 measured reflections	l = −7→7

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.034	H-atom parameters constrained
$wR(F^2)$ = 0.102	$w = 1/[\sigma^2(F_o^2) + (0.0654P)^2 + 2.4853P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.001

1572 reflections	$\Delta\rho_{max}$ = 1.40 e Å^{-3}
92 parameters	$\Delta\rho_{min}$ = −1.17 e Å^{-3}
57 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Hg1	0.92942 (2)	0.7500	0.65028 (6)	0.0427 (2)	
Br1	0.913 (4)	0.606 (5)	0.471 (9)	0.058 (2)	0.142 (1)
Cl1	0.9142 (14)	0.6012 (19)	0.441 (3)	0.058 (2)	0.858 (1)
Br2	0.8251 (2)	0.7500	0.9091 (3)	0.0849 (8)	0.921 (4)
Cl2	0.821 (8)	0.7500	0.853 (14)	0.0849 (8)	0.079 (4)
Br3	1.06769 (13)	0.7500	0.7582 (4)	0.0631 (7)	0.795 (4)
Cl3	1.0543 (16)	0.7500	0.818 (5)	0.0631 (7)	0.205 (4)
N1	0.59356 (19)	0.5290 (3)	0.4047 (7)	0.066 (2)	
C1	0.6519 (3)	0.5275 (4)	0.5418 (6)	0.078 (3)	
H1	0.6479	0.4924	0.6583	0.094*	
C2	0.7161 (2)	0.5786 (4)	0.5046 (7)	0.057 (2)	
H2	0.7551	0.5776	0.5963	0.068*	
C3	0.7221 (2)	0.6311 (4)	0.3304 (7)	0.059 (2)	
H3	0.7651	0.6653	0.3056	0.071*	
C4	0.6638 (3)	0.6325 (4)	0.1933 (6)	0.055 (2)	
H4	0.6678	0.6677	0.0768	0.067*	
C5	0.5995 (2)	0.5815 (4)	0.2305 (7)	0.064 (2)	
H5	0.5605	0.5824	0.1388	0.077*	
C6	0.5273 (3)	0.4739 (4)	0.4394 (11)	0.075 (3)	
H6A	0.5407	0.4170	0.5085	0.090*	
H6B	0.5050	0.4572	0.3118	0.090*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Hg1	0.0363 (3)	0.0426 (3)	0.0491 (3)	0.000	0.00310 (16)	0.000
Br1	0.0424 (11)	0.064 (3)	0.067 (7)	−0.0014 (15)	−0.001 (4)	−0.031 (4)
Cl1	0.0424 (11)	0.064 (3)	0.067 (7)	−0.0014 (15)	−0.001 (4)	−0.031 (4)
Br2	0.0753 (12)	0.1119 (13)	0.0676 (17)	0.000	0.0385 (16)	0.000
Cl2	0.0753 (12)	0.1119 (13)	0.0676 (17)	0.000	0.0385 (16)	0.000
Br3	0.0350 (12)	0.0791 (11)	0.0751 (19)	0.000	−0.0030 (9)	0.000
Cl3	0.0350 (12)	0.0791 (11)	0.0751 (19)	0.000	−0.0030 (9)	0.000
N1	0.031 (3)	0.096 (5)	0.072 (4)	−0.011 (4)	−0.002 (3)	0.031 (4)
C1	0.057 (5)	0.104 (6)	0.075 (6)	−0.010 (5)	−0.009 (4)	0.033 (5)
C2	0.025 (4)	0.069 (5)	0.076 (5)	−0.003 (3)	−0.008 (3)	0.017 (4)
C3	0.041 (4)	0.066 (5)	0.070 (5)	−0.009 (4)	0.004 (4)	0.016 (4)
C4	0.049 (5)	0.054 (4)	0.063 (4)	−0.015 (4)	0.004 (4)	0.010 (4)
C5	0.047 (4)	0.075 (5)	0.070 (5)	−0.011 (4)	0.004 (4)	0.022 (4)
C6	0.087 (6)	0.066 (5)	0.073 (5)	0.018 (5)	−0.008 (5)	−0.008 (4)

Geometric parameters (Å, °)

Hg1—Cl2	2.37 (11)	C1—H1	0.93
Hg1—Br1	2.41 (5)	C2—C3	1.39
Hg1—Br1^{i}	2.41 (5)	C2—H2	0.93
Hg1—Cl3	2.50 (3)	C3—C4	1.39
Hg1—Br2	2.548 (3)	C3—H3	0.93
Hg1—Cl1^{i}	2.571 (19)	C4—C5	1.39
Hg1—Cl1	2.571 (19)	C4—H4	0.93
Hg1—Br3	2.584 (3)	C5—H5	0.93
N1—C1	1.39	C6—C6^{ii}	1.476 (6)
N1—C5	1.39	C6—H6A	0.97
N1—C6	1.448 (6)	C6—H6B	0.97
C1—C2	1.39		
Cl2—Hg1—Br1^{i}	101 (2)	C1—N1—C5	120.0
Cl2—Hg1—Br1	101 (2)	C1—N1—C6	120.4 (4)
Br1^{i}—Hg1—Br1	119 (4)	C5—N1—C6	119.6 (4)
Cl2—Hg1—Cl3	119 (3)	N1—C1—C2	120.0
Br1^{i}—Hg1—Cl3	109.1 (17)	N1—C1—H1	120.0
Br1—Hg1—Cl3	109.1 (17)	C2—C1—H1	120.0
Br1^{i}—Hg1—Br2	104.4 (16)	C3—C2—C1	120.0
Br1—Hg1—Br2	104.4 (16)	C3—C2—H2	120.0
Cl3—Hg1—Br2	110.9 (6)	C1—C2—H2	120.0
Cl2—Hg1—Cl1^{i}	103.0 (16)	C4—C3—C2	120.0
Br1—Hg1—Cl1^{i}	115.7 (11)	C4—C3—H3	120.0
Cl3—Hg1—Cl1^{i}	109.7 (7)	C2—C3—H3	120.0
Br2—Hg1—Cl1^{i}	106.9 (6)	C3—C4—C5	120.0
Cl2—Hg1—Cl1	103.0 (16)	C3—C4—H4	120.0
Br1^{i}—Hg1—Cl1	115.7 (11)	C5—C4—H4	120.0
Cl3—Hg1—Cl1	109.7 (6)	C4—C5—N1	120.0
Br2—Hg1—Cl1	106.9 (6)	C4—C5—H5	120.0
Cl1^{i}—Hg1—Cl1	112.7 (13)	N1—C5—H5	120.0
Cl2—Hg1—Br3	129 (3)	N1—C6—C6^{ii}	110.8 (6)
Br1^{i}—Hg1—Br3	104.6 (16)	N1—C6—H6A	109.5
Br1—Hg1—Br3	104.6 (16)	C6^{ii}—C6—H6A	109.5
Br2—Hg1—Br3	121.16 (9)	N1—C6—H6B	109.5
Cl1^{i}—Hg1—Br3	104.7 (6)	C6^{ii}—C6—H6B	109.5
Cl1—Hg1—Br3	104.7 (6)	H6A—C6—H6B	108.1
C6—N1—C1—C2	178.2 (3)	C1—N1—C6—C6^{ii}	87.7 (9)
C6—N1—C5—C4	−178.2 (3)	C5—N1—C6—C6^{ii}	−94.1 (9)

Symmetry codes: (i) *x*, −*y*+3/2, *z*; (ii) −*x*+1, −*y*+1, −*z*+1.

Fig. 1